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AMINOMETHYLPHOSPHONIC ACID

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Aminomethylphosphonic acid is prepared on the basis of the following reactions:

$$ClCH_2PO(OC_2H_5)_2 \xrightarrow[H_2O]{NH_3} H_2NCH_2P(O)(OH)OC_2H_5 \xrightarrow[H_2O]{HCl} H_2NCH_2P(O)(OH)OH$$

Aside from the method under description, the relevant literature on methods of preparation comprises patent description (1) for preparing aminomethylphosphonic acid from methylol derivatives of carboxylic acid amides and phosphorus trichloride, and the work of Chavane on the phthalimide synthesis of this acid. Neither of these methods is a good procedure for the preparation of aminomethylphosphonic acid.

The present method for the preparation of aminomethylphosphonic acid (3) is based on the reaction between aqueous ammonia and the ethyl ester of chloromethylphosphonic acid. The monoethyl ester of aminomethylphosphonic acid thus formed readily hydrolyzes when heated with hydrochloric acid. The yield amounts to 48-50% computed on the basis of chloromethylphosphonic acid. Instead of the ester of chloromethylphosphonic acid, the ethyl ester of iodomethylphosphonic acid can be employed with equal success. The synthesis consists of two stages.

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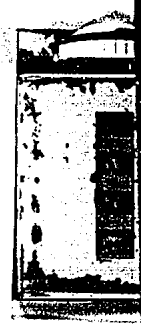


Description of the Synthesis

1. Monoethyl Ester of Aminomethylphosphonic Acid

Fifty g of the ethyl ester of chloromethylphosphonic acid and 240 ml of 25% ammonia are heated in several sealed tubes at 150° for one hour. In order to prevent explosion of the tubes, it is best to place them in either an autoclave or a bomb having an adequate counterpressure (25-30 atm at 150°). After the tubes have been opened, their contents are evaporated on a water bath. The residue, a sirupy liquid, is dissolved in a small quantity of water and the solution is shaken with 35 g of freshly prepared moist silver oxide. The residue of silver chloride and the excess silver oxide are filtered off, the silver removed from the filtrate with hydrogen sulfide, the silver sulfide filtered off, and both the filtrate and the wash water evaporated to constant volume. The residue, a yellowish sirup, is dissolved in 95% alcohol. An alcoholic solution of 54 g of aniline (2 moles of aniline per mole of starting substance) is added to the solution. After prolonged standing, the aniline salt of the monoethyl ester of aminomethylphosphonic acid which has formed is filtered off with vacuum suction and washed with alcohol. 26-27 g are obtained of a colorless crystalline substance with a melting point of 230°. The substance has the composition:

$$[NH_2CH_2PO(OC_2H_5)OH]_2 \cdot C_6H_5NH_2.$$

The substance is freed from aniline by recrystallization from aqueous alcohol or by heating the aniline salt to 100° under vacuum (2-4 mm residual pressure) for a period of several hours until constant weight.

When the aniline salt is recrystallized from the aqueous alcohol (see note), 16-17 g of acidic ethyl ester of aminomethylphosphonic acid with a melting point of 240° are obtained

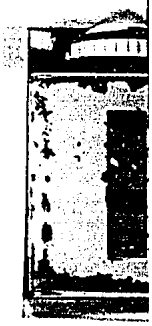
2. Aminomethylphosphonic Acid

Fifty g of hydrochloric acid (1 : 1) are added to 3 g of the monoethyl ester of aminomethylphosphonic acid and the mixture is heated in sealed tubes to 120-140° for 3 hours. After the tubes have been opened, the solution is evaporated to dryness on a water bath, the residue dissolved in a small quantity of water, and some alcohol added to the mixture for the precipitation of the product. The precipitated crystals are recrystallized from a water-alcohol mixture and dried. 2.25 g of a colorless crystalline substance with a melting point of 310° are obtained. The yield is 94% of the theoretical.

Properties

The monoethyl ester of aminomethylphosphonic acid is a colorless crystalline substance having a melting point of 240° It is very soluble in water and hot alcohol, but insoluble in benzene, ether, and other organic solvents. Aqueous solutions have a neutral reaction on litmus

Aminomethylphosphonic acid consists of colorless crystals melting with decomposition at 310°. It is very soluble in water, but poorly soluble even in hot alcohol. It is insoluble in organic solvents. Its aqueous solution has a neutral reaction, and in the presence of either phenolphthalein or thymolphthalein, it titrates like a monobasic acid. In the presence of alizarine red, the inner salt of the aminoacid is also titrated .



Note

For recrystallization, aniline salt is treated with 100-120 ml of 96% hot ethyl alcohol; the salt then partially dissolves. Water is added by drops to the hot solution until the solution is clear. The solution is then filtered and, after cooling, an excess of absolute ethyl alcohol is added. The precipitated crystals are then filtered.

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